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EXPERIMENTAL INVESTIGATION OF ULTRA-HIGH VACUUM ADHESION AS RELATED TO THE LUNAR SURFACE

FIRST QUARTERLY PROGRESS REPORT
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1.0 INTRODUCTION

1.1 General

This report presents a summary of the work accomplished during the period July through September 1964 on the study of the ultra-high vacuum frictional-adhesional behavior of silicates as related to the lunar surface. In addition, because this is the first report since initiation of the study, a brief review is given of present ideas as to the nature of friction-adhesion, of previous work relating to this study, and of the physical nature of silicates.

1.2 Purpose and Importance of Program

The primary purpose of this program is to obtain quantitative experimental data concerning the ultra-high vacuum adhesional-frictional behavior of the materials which may presently exist at the lunar surface, and between these and engineering materials which may be placed upon the surface. Additional purposes are to analyze these data with regards to the possible reactions of granular lunar materials to engineering operations, and to investigate means by which the problems, if any, posed by these reactions may be minimized.

The importance of this program is that adhesional-frictional phenomena may pose serious problems to lunar surface operations, and as yet very little pertinent information is available.

1.3 Approach

The approach used in the first phase of this study (the approach for subsequent phases will be detailed at a later time) is to obtain quantitative data relating to the adhesion force as a function of load force, temperature, type of silicate,

crystalline orientation, and surface preparation; and then to use these data to analyze the possible behavior of silicates at the lunar surface and the problems this may pose to lunar operations.

Single crystals of each mineral are used since this allows one to obtain understanding as to the basic physics of silicate behavior in ultra-high vacuum. Load forces are applied by means of an electromagnet which is placed outside the vacuum system, thereby reducing vacuum problems. Since the few available data indicate the silicate adhesion forces may be small, the approach used in this study is to use as sensitive an adhesion measuring apparatus as possible. The decision has been made to use microbalance techniques. Details of the experimental techniques employed are given in following sections.

2.0 SUMMARY OF PREVIOUS WORK

2.1 Concepts of Friction and Adhesion

The generally accepted theory of friction is the so-called "adhesional" of "weld-junction" theory developed by Bowden and many others. It is so well known that only the barest outline of it need be given. This theory states that when two surfaces are placed in contact the load is borne by a few contacting surface asperities. The high pressures developed cause plastic flow until the true area of contact is sufficient to support the load. At these points of contact atoms of one surface are within range of the attractive forces of the atoms of the other surface and adhesive bonds are formed. The strength of these bonds depends upon whether the interactions are between atoms of the bulk material or between surface contaminents, being greatest in the absence of contamination. This leads immediately to the concept that friction is produced by the force required to

shear these bonds.

This theory, developed principally for metals, has been applied with a reasonable degree of success to the non-metals as well (see for instance the list of references to this work given by Walton (1962)). Regardless of the success achieved to date, the theory remains as an incomplete, and in certain cases a somewhat dubious, solution to the friction (and adhesion) question. This is not completely unexpected, however, since friction phenomena relate to surface interactions and bulk reactions, many of which are not well understood. It is worth enumerating (and discussing where applicable) some of the uncertainties involved.

An important concept in the "adhesion" theory is that of plastic flow. By assuming plastic flow at the few contacting asperities, and that the yield and shear strengths of the material remain constant, it is possible to satisfy Amonton's Laws (which are themselves, it should be noted, not of universal validity). However, it has been noted by Archard (1961) and others that the invoking of plastic flow is not necessary to explain these Laws. According to Archard, if one permits multiple contacts (significantly greater than three), Amonton's Laws can be satisfied with purely elastic asperity deformation. The possible reality of this effect in certain cases has received partial verification from the work of Dyson and Hirst (1954). Nevertheless, it does not appear that Archard's mechanism could be generally operative, since it depends upon what could be called a certain "regularity" (in height) in the surface roughness. However, it is possible that under certain conditions of surface preparation (for instance during polishing) such regularity could well be produced. This possibility points out one variable of frictional phenomena not explicitly considered in the adhesion theory: surface

preparation.

There are two other important factors relating to surface preparation. These are the degree of surface roughness and the effect of surface preparation upon the physico-chemical state of the surface and near surface layers. The significant frictional role played particularly by extreme surface roughness is obvious. For most engineering applications the roughness is such that it is generally disregarded as a separate frictional term. However, for the understanding of soil behavior it becomes of considerable importance. Significant physico-chemical changes can be produced by polishing. In particular, a glassy or cryptocrystalline state can be formed in the immediate surface layers. This effect, though of considerably less importance for silicates than for metals, must be taken into account. Its importance to frictional phenomena will become evident shortly when the roles played by crystalline structure and the nature and types of atomic bonding are discussed.

One consequence of the "adhesion" theory of friction is that a finite force should be required to separate contacting surfaces. This force has indeed been detected, but only under certain conditions. One such condition is vacuum where with suitable surface cleaning many, but not all, materials have been found to adhere, some quite strongly. Unfortunately, despite these findings, essentially no quantitative data are available. Another condition under which materials on occasion have been found to adhere is when one or both is of sufficiently low hardness (or the load force sufficiently high) for bulk plastic flow to occur. The lack of observable adhesion in many cases has been ascribed variously as being due to the action of released elastic stresses, the presence of oxide layers, the presence of absorbed gases, and/or the general incompatibility of the materials comprising the surfaces.

It is reasonable to expect that for contacting surfaces, unless the materials are quite soft, upon release of load some degree of elastic recovery will occur. This elastic recovery will tend to break junctions formed during the prior loading. The introduction of elastic recovery brings a whole host of new variables into the frictional-adhesional phenomena. These relate directly to the bulk physical properties of the material such as hardness, the elastic and plastic parameters, and strength (tensile, yield, shear, compressional, and ultimate); and to such considerations as junction geometry (relating particularly to stress concentrations) and temperature.

It has been found (see for instance Walton (1962)) that elastic recovery is not sufficient to account for the often noted lack of observable adhesion. Calculations indicate that if this were the only factor operative then easily measurable adhesion should remain in essentially all cases, at zero load. This difficulty led in part to consideration of the role played by oxide films (on metals: metallic friction historically having received most attention). One possible role of oxide films had been known for some time, the argument being that increased elastic recovery occurs in the presence of an oxide layer (harder than underlying metal) and hence in the presence of such a layer the adhesion, under zero load, would be much less than that of the pure metal alone. However, a new possible role was uncovered, this pertaining to the type of atomic bonds formed across the interface. The metallic bond being highly non-directional can tolerate a significant amount of atomic mis-match across the interface and yet still produce strong adhesion. The oxide bonds, on the other hand, are considerably more directional, and hence unless a perfect or near-perfect atomic match were made across the interface the resultant adhesion may be quite weak. A similar role has been postulated for adsorbed gas films. These films when present are believed to keep the materials

separated to the extent that the normal atomic bonding forces cannot come into play across the interface. The only remaining active forces are then those between the adsorbed gas molecules which are of the weak Van der Walls type. These concepts, which appear to be valid, add additional variables to frictional phenomena: atomic bond type(s) present and acting (directionally and strength) and crystalline structure. The importance of physico-chemical alterations in surface and near-surface structure, noted earlier, which may be produced during surface preparation now becomes apparent.

These concepts led to the introduction of the "work of adhesion" by Rabinowitz (1961), a quantity associated with the surface free energy which in turn is some function of the atomic bond types, crystal structure, and crystalline orientation. This concept and the problems associated with it have been discussed by Spalvins and Keller (1962). Experimental data relating to this concept and indicating the importance of the contained variables have been obtained by Spalvins and Keller (1962), Riesz and Weber (1962), Duwell (1962), Steijn (1963), Roshon (1964) and others. Unfortunately, a considerable amount of work remains to be done before a detailed understanding of these variables can be achieved.

The various variables of frictional-adhesional phenomena are listed, for convenience, in Table 1. Though these may appear to cover the entire list of possible variables, there is no assurance that this is actually the case. Also, it is difficult at present to weigh the relative importance of each.

TABLE I

Variables Relating to Frictional - Adhesional Phenomena

Atomic Related Variables

- Atomic bond type - strength, directionality

Crystalline structure

Interface Related Variables

Roughness (including roughness regularity)

Contamination (type and degree of)

Junction Geometry

Crystalline Orientations

Chemical Composition

Physico-Chemical Surface State

Bulk Related Variables

Hardness

Elastic & Plastic Properties

Strength Properties

Miscellaneous

Temperature

2.2 Experimental Evidence for Silicate Adhesion

There is as yet no direct evidence as to the composition of the lunar surface. However, through the indications provided by terrestrial and meteoritic materials it is generally believed that the surface is composed primarily of silicates. Unfortunately, there is at present no great abundance of data relating to the adhesion of silicates.

The earliest work of interest appears to be that by Tomlinson (1928, 1930) and by Stone (1930). Tomlinson measured adhesion between glass and quartz (not strictly, at least historically, a silicate) balls and fibers, detecting forces between the spheres as large as one gram. Tomlinson's results, particularly his interpretations of the adhesion as being atomic were challenged by Stone, but apparently no satisfactory resolution of their differences was achieved. It should be noted, however, that the work was done in air and even though careful cleaning techniques were used, a reasonably large amount of surface contamination, particularly adsorbed water, was undoubtedly present. More recently, Harper (1955) performed adhesion experiments with quartz spheres, in air, finding adhesional forces as large as 0.15 gm. Though he presented convincing arguments that these forces were not due to surface charging, it is likely that at least a mono-layer of adsorbed water was present and hence it is difficult to say what fraction of this adhesion force was indeed due to atomic quartz-quartz interactions. A few additional experiments with micas have been performed but these are of no particular use to the present problem.

It has not been until the last few years that the first experiments in vacuum were performed. These have demonstrated the presence of silicate adhesion. Salisbury et al (1964) conducted experiments with polycrystalline silicate powders at a vacuum in the mid 10^{-10} mm Hg range. They found adherence of the powder grains

($\approx 5\,\mu$ in diameter) and made a rough calculation that the adhesion force was $\approx 2 - 3 \times 10^{-7}$ gm. In these experiments there was no high temperature or other (e.g. ionic-electronic) outgassing attempted and the adhesion was that under essentially zero prior load. This work was followed by that of Stern and Johnson (1964) who studied larger grains (up to $140\,\mu$ in diameter) at pressures of 6.3×10^{-10} to 1.3×10^{-9} mm Hg and with one day outgassing at about 100°C. They found that the force of adhesion (with no prior loading) increased with particle size, being in excess of $\approx 30\,\mu$ g for the larger particles. They noted that if prior loading had been used the adhesion force may have been significantly greater than this. Additional experiments have been performed by Halajian (1964), also upon powders, but of about $40\,\mu$ in diameter. The pressures obtained were in the high 10^{-10} mm Hg range and the system was maintained continuously at 200°C. From Halajian's results one can make a rough calculation as to the adhesion force, utilizing the method applied by Salisbury, finding that it was at least $30\,\mu$ g.

Much more needs be done before even a reasonable understanding of the ultra-high vacuum behavior of silicates can be reached. In particular, quantitative data under controlled conditions should be obtained. This involves discarding the polycrystalline powders previously used and utilizing instead single crystal samples of particular silicate minerals. With these, effects of crystalline orientation can be investigated and quantitative data concerning load vs adhesion force and temperature vs adhesion force can be obtained. Also, improved surface outgassing should be realized by raising the samples to temperatures not very much less than their melting point, or by using ion-electron bombardment.

3.0 THE SILICATES

It is of interest, since the majority of effort during this study is concentrated upon the silicates, to outline briefly the physical nature of silicate systems such as occur in terrestrial and meteoritic materials.

The silicates are as a whole highly stable structures. The basic building unit of all silicates is the silica tetrahedron consisting of a silicon atom (at the center) surrounded by four oxygens (at the vertices). The silicon-oxygen bond is intermediate between a pure covalent and pure ionic type. The wide diversity within the silicate family can be explained by the varying degrees to which these oxygen atoms are shared by a second silicon, also by the fact that there are a number of other atoms which can either substitute for the silicon (such as aluminum) or can enter into the general lattice (such a potassium, sodium, calcium, barium, aluminum, and the OH^- radical). On the basis of oxygen sharing the silicates are generally grouped into six classes: independent tetrahedral groups (the orthosilicates); double tetrahedral structures (dimers); ring structures; chain structures; sheet structures; and three dimensional networks. A wide variety of mineral types are found within each class due to the introduction into the lattice of various different atoms.

The general characteristics of each class are as follows:

(1) Independent tetrahedral groups

No oxygens are shared and each silica tetrahedron is in this sense independent of all others. The crystal integrity is maintained by bonding between the oxygens and cations other than silicon. Examples of this type of structure are olivine (an important constituent of meteorites) and the epidote group of minerals.

(2) Double tetrahedral structures

The tetrahedra occur in pairs with a single oxygen per pair being shared. Each pair is separated from all other pairs, the remaining oxygens bonding with cations other than silicon. An example of this type of structure is shown by hemimorphite.

(3) Ring structures

Two oxygens atoms per tetrahedron are shared. The tetrahedra form rings containing two, three, four or six tetrahedra per ring. The remaining oxygens bond with cations other than silicon. An example of this class is beryl (six tetrahedra per ring).

(4) Chain structures

(a) Single chain

Two oxygens per tetrahedron are shared and the tetrahedra are joined into chains of "infinite" extent. The chains normal to their length are bonded by means of linkages between the remaining oxygens and cations other than silicon. An example of this type of structure is given by the pyroxene group of minerals, relatively important constituents of terrestrial igneous rocks, particularly the more basic varieties, and meteorites.

(b) Double chains

The tetrahedra share alternately two and three oxygens forming double linked chains of "infinite" extent. The chains normal to their length are bonded by means of linkages between the remaining oxygens and cations other than silicon. An example of this type of structure is given by the amphibole group of minerals.

(5) Sheet structures

Three oxygen atoms are shared per tetrahedron, the remaining oxygen bonding with cations other than silicon. The silicon bonded oxygens form parallel planes of "infinite" extent. An outstanding example of this type of structure is given by the micas, a relatively common constituent of terrestrial igneous rock.

(6) Three-dimensional networks

All oxygens of each tetrahedra are shared with adjacent tetrahedra. The diversity of minerals in this class results from the replacement of some of the silicon atoms and the introduction of additional atoms into the structure to maintain charge neutrality. An example of this type of structure is given by the feldspars, most important rock and meteorite constituents.

The silicates are characterized by significant variation in bond strengths and bond types. The ionic-covalent type bonds dominate, and within any given mineral two or more bond types (within the ionic-covalent extremes) are common. Cleavage, a characteristic feature of many silicates, is thus explained as being due to the presence of weaker (ionic) bonding in certain directions; also of course in part to the geometric configurations of the atoms in the lattice.

It is of interest, in the light of these comments, to consider what the ultra-high vacuum frictional-adhesional behavior of silicates may be. First, since the silicate bonding varies within degrees of being more ionic or more covalent, and several different types of bonds can exist in a single mineral crystal, this implies that the strength of adhesion is sensitive both to type of crystal and crystal orientation.

Second, ionic-covalent bonds are more directional than metallic bonds and this implies that to the degree to which this difference is important the adhesional forces between silicates should be significantly less than those between metals (it also implies a decided crystal orientation sensitivity). Finally, the relative hardness and brittleness of silicates also implies some significant degree of elastic recovery upon removal of load. This also indicates that silicate adhesion may be less than that of the metals, at least for those metals found to adhere.

In any comprehensive treatment of the ultra-high vacuum frictional-adhesional behavior of silicates, representatives from each of the classes noted in this section should be investigated. However, for the present study it is of more importance to investigate the common silicate minerals such as may predominate at the lunar surface. This is used as the primary criterion for sample choice in the present study. The samples chosen for study, as well as the reasons for their choice, are discussed in a following section. It is worthwhile noting, however, that the above structural groups which contain the most common igneous and meteoritic-contained silicates are: the independent tetrahedral groups, the chain structures, the sheet structures, and in particular the three dimensional networks.

4.0 INSTRUMENTATION

4.1 Vacuum System

The vacuum system for this study has been assembled during this quarter. It consists of four major parts: forepump, cold trap, ion pump, and the experimental chamber. This system is shown schematically in Figure 1. The mechanical forepump provides the initial pumping down to a pressure of 10^{-3} - 10^{-4} mm Hg. The cold

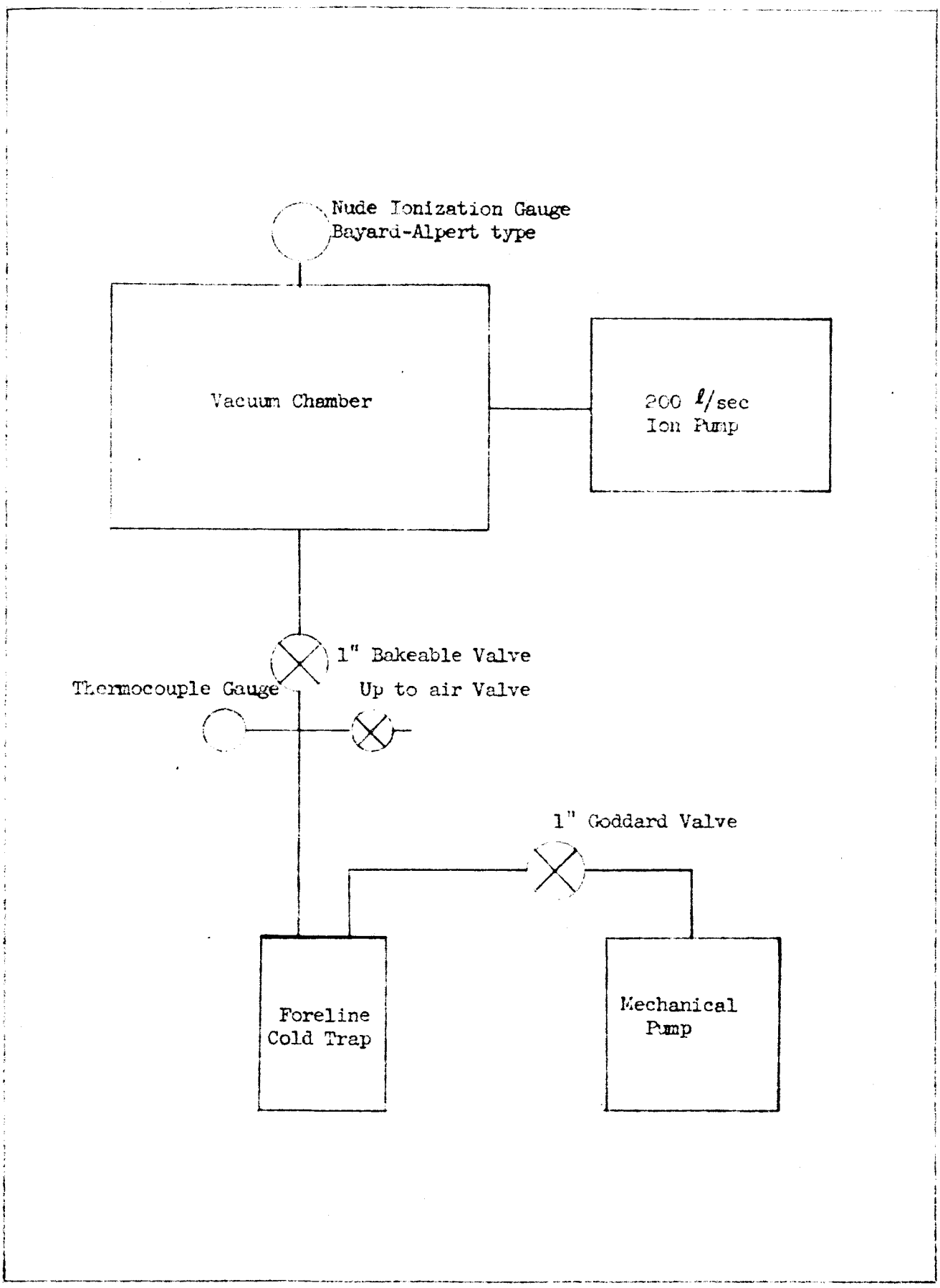

Figure 1 Schematic of Vacuum System

trap (liquid nitrogen) is utilized to prevent migration of oil vapors into the experimental chamber. It is degreased prior to every pumpdown. The basic unit for obtaining ultra-high vacuum is the ion pump. It has a pumping speed of 200 liters sec^{-1} at a pressure of 10^{-8} mm Hg. Its speed decreases slowly through the 10^{-9} and mid 10^{-10} mm Hg. range: it has a rated ultimate in the 10^{-11} mm Hg range. Preliminary pumpdowns, without the presence of the complete experimental apparatus and with only moderate bakeout, have given pressures of about 2×10^{-10} mm Hg as read by a "nude" Bayard-Alpert ionization gage. Good agreement has been found between readings given by this gage and the pressure indicated by the ion pump current. The experimental chamber, along with the ion pump itself, constitutes the ultra-high vacuum part of the system. This section is separated from the fore-pump and cold trap by means of an ultra-high vacuum bakable valve. This valve is closed during operation of the ion pump.

The ultra-high vacuum section is of all-metal construction, principally 304 Stainless, with the vacuum seals being made by means of copper and gold gaskets. The chamber itself consists of a six inch (diameter) tee and a six inch cross upon which are mounted the load application and adhesion measuring systems (see Figure 2). Two viewing ports are provided to permit observation of the experiment. One linear motion feedthrough and an eight pin electrical feedthrough are installed on the top flange of the tee (see Figures 2 and 3). The bottom flange of the tee contains the sample holder (see Figures 2, 3, and 4) and provides means whereby an electromagnet and a heater can be applied externally to provide load force and high temperature outgassing respectively.

4.2 Load Application System

The load application system provides the load force to press the samples together. It is shown best in Figure 4. The system employs an electromagnet outside the

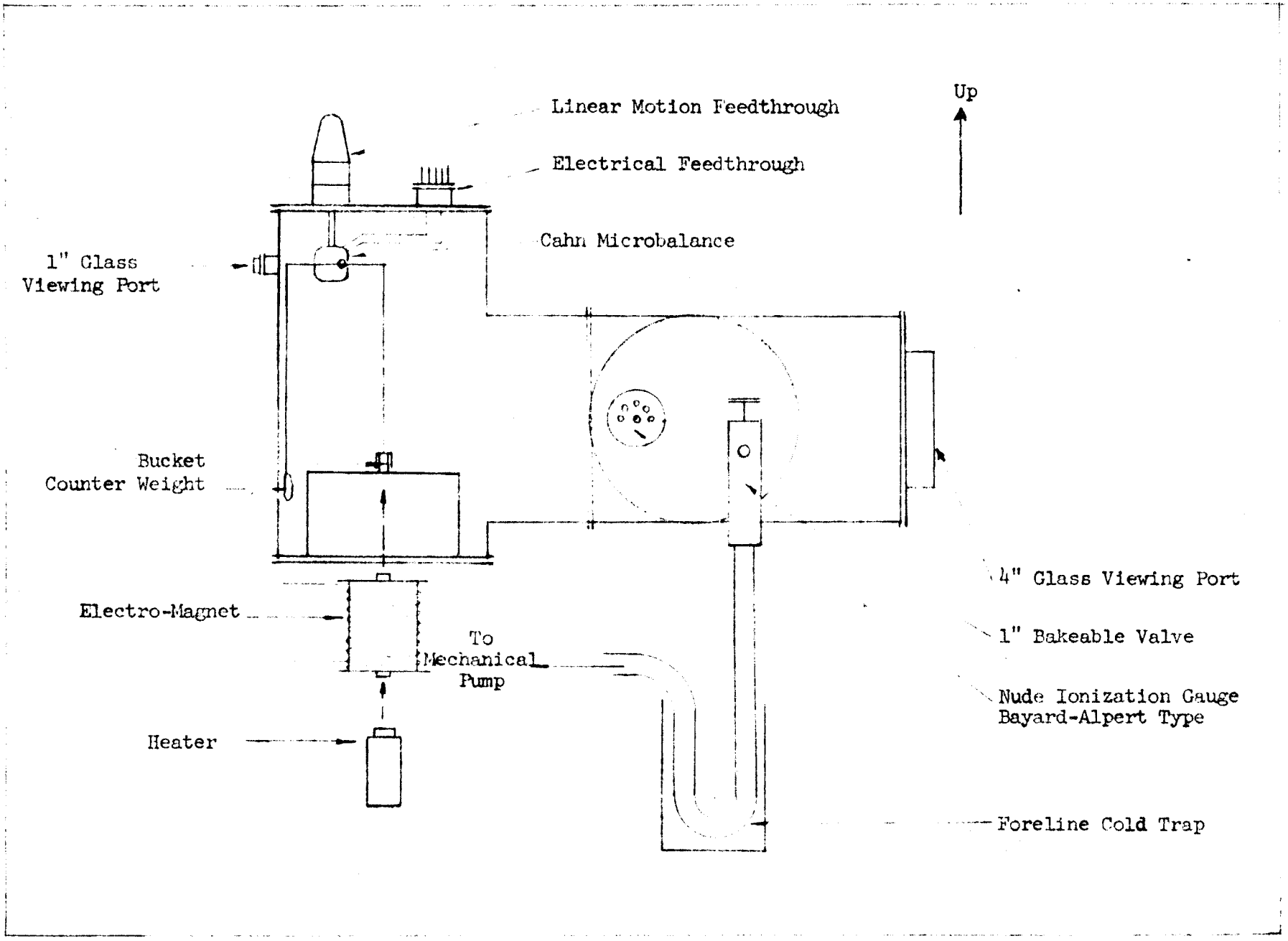

Figure 2 Adhesion Apparatus

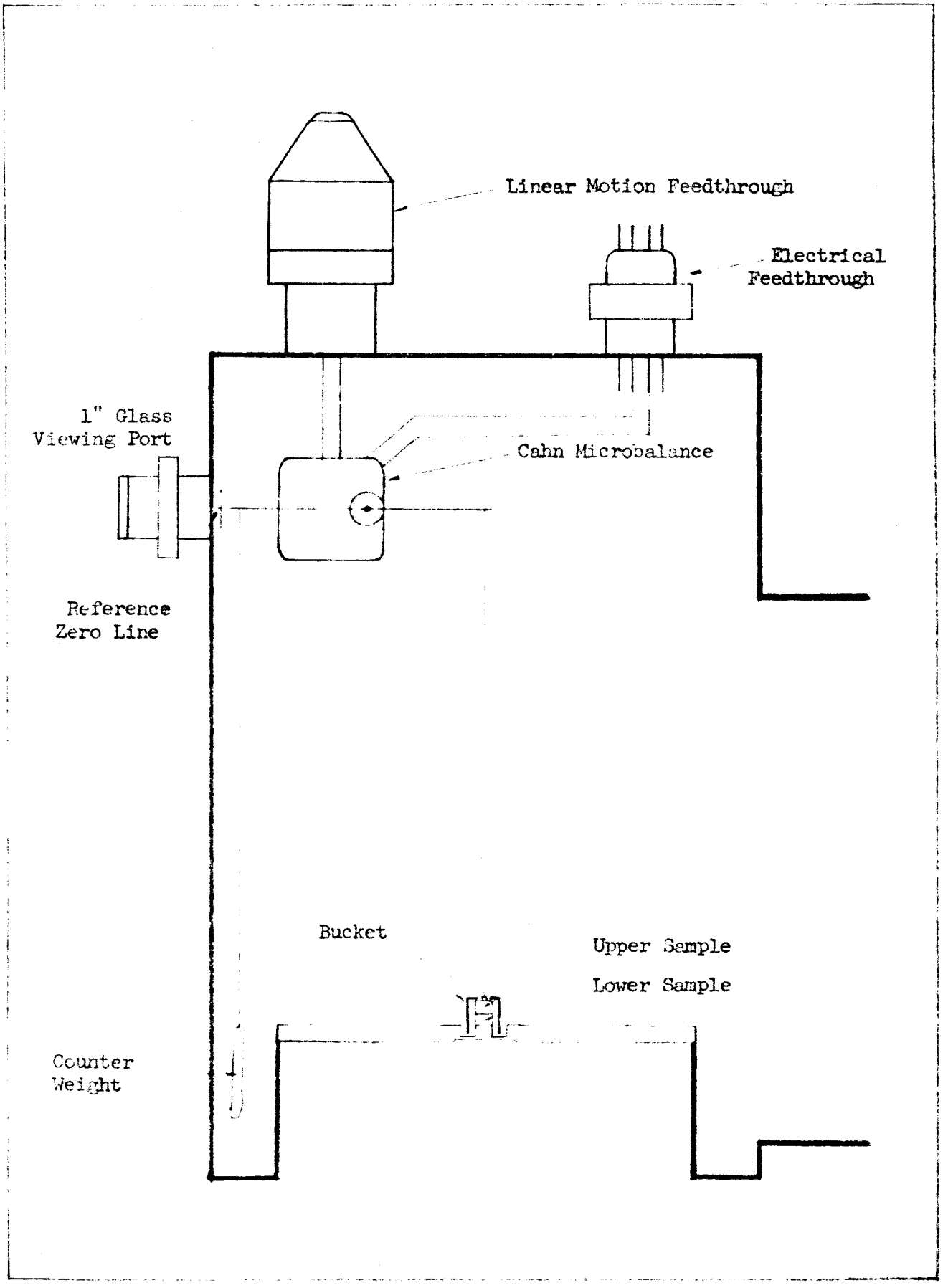

Figure 3 Adhesion Measuring System

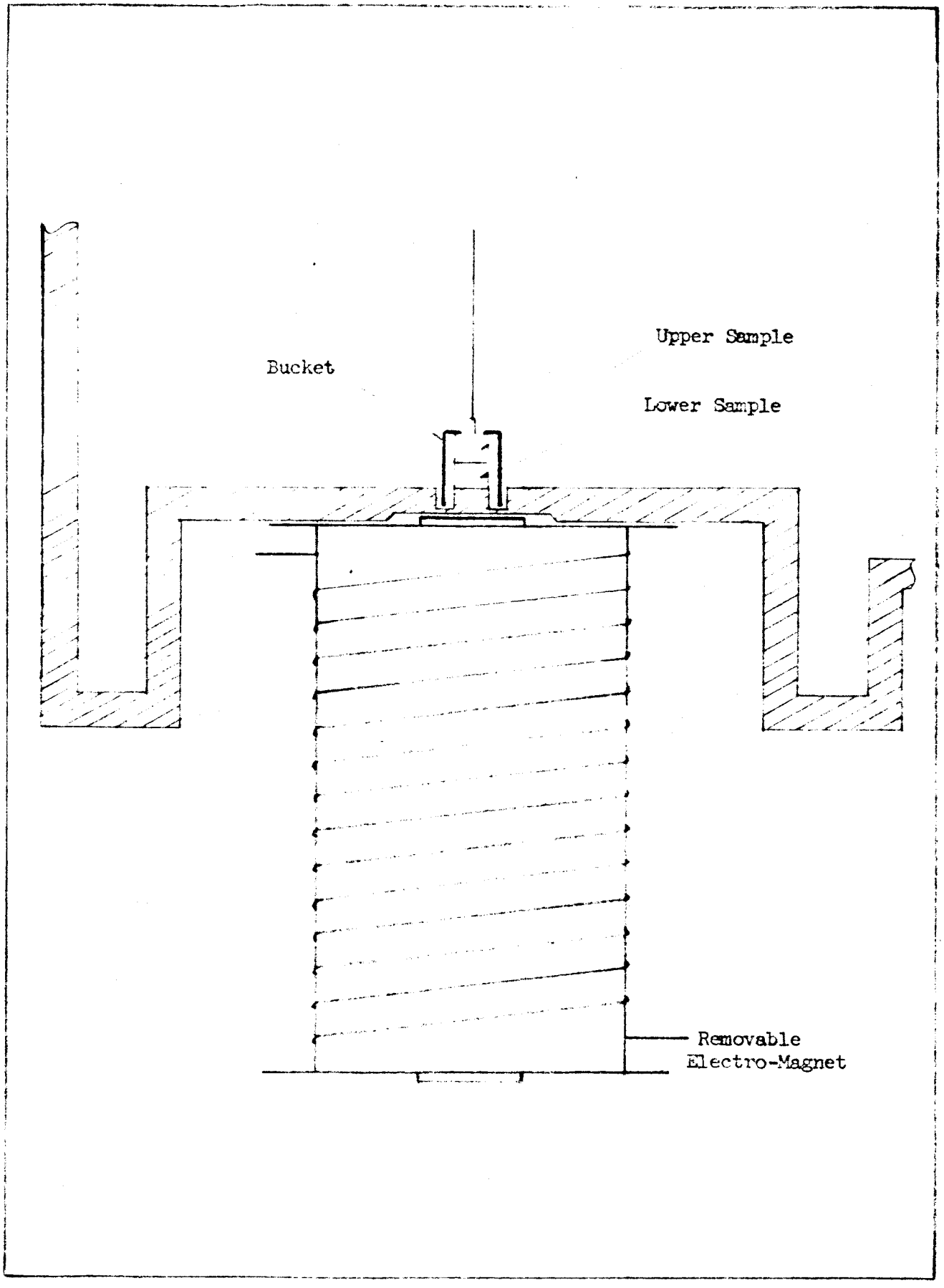

Figure 4 Load Application System

vacuum chamber and a steel bucket inside the chamber. The bucket, resting on the upper sample, has been designed to meet the conditions that its weight should not overload the adhesion measurement system, that it at no time contacts any other components, that load forces up to $\approx$1000 gms. can be applied, and that the geometric configuration be such that the bucket remains stable (in orientation) during operation and allows observation of the contacting surfaces. These conditions have been met successfully with the exception that the bucket weight ($\cong$ 0.9 gm) has resulted in some loss of sensitivity in the adhesion measuring system. This is discussed in the following section.

A battery power supply has been constructed for use with the electromagnet. Several calibrations of load force as a function of current drawn have been made. A typical curve is shown in Figure 5. Here, load force is measured by means of Chatillon precision mechanical springs. The scatter in the points at higher current is due primarily to force reading difficulties caused by an overly compressed spring scale. This difficulty has been removed by obtaining a spring with a more expanded scale in this range. No particular difficulties have been encountered yet with hysteresis effects. Should these appear, high temperature (above Curie point) bakeout of bucket and core will be used prior to each load application.

4.3 Adhesion Measuring System

The basic unit for measuring the force of adhesion is a modified Cahn microbalance. This balance is essentially a galvanometer movement. Current through the meter movement coil (suspended in a magnetic field) applies torque to the balance arm, which along with the coil is supported by means of an elastic metal fiber. The adhesion force is then measured as the current which must be passed through the

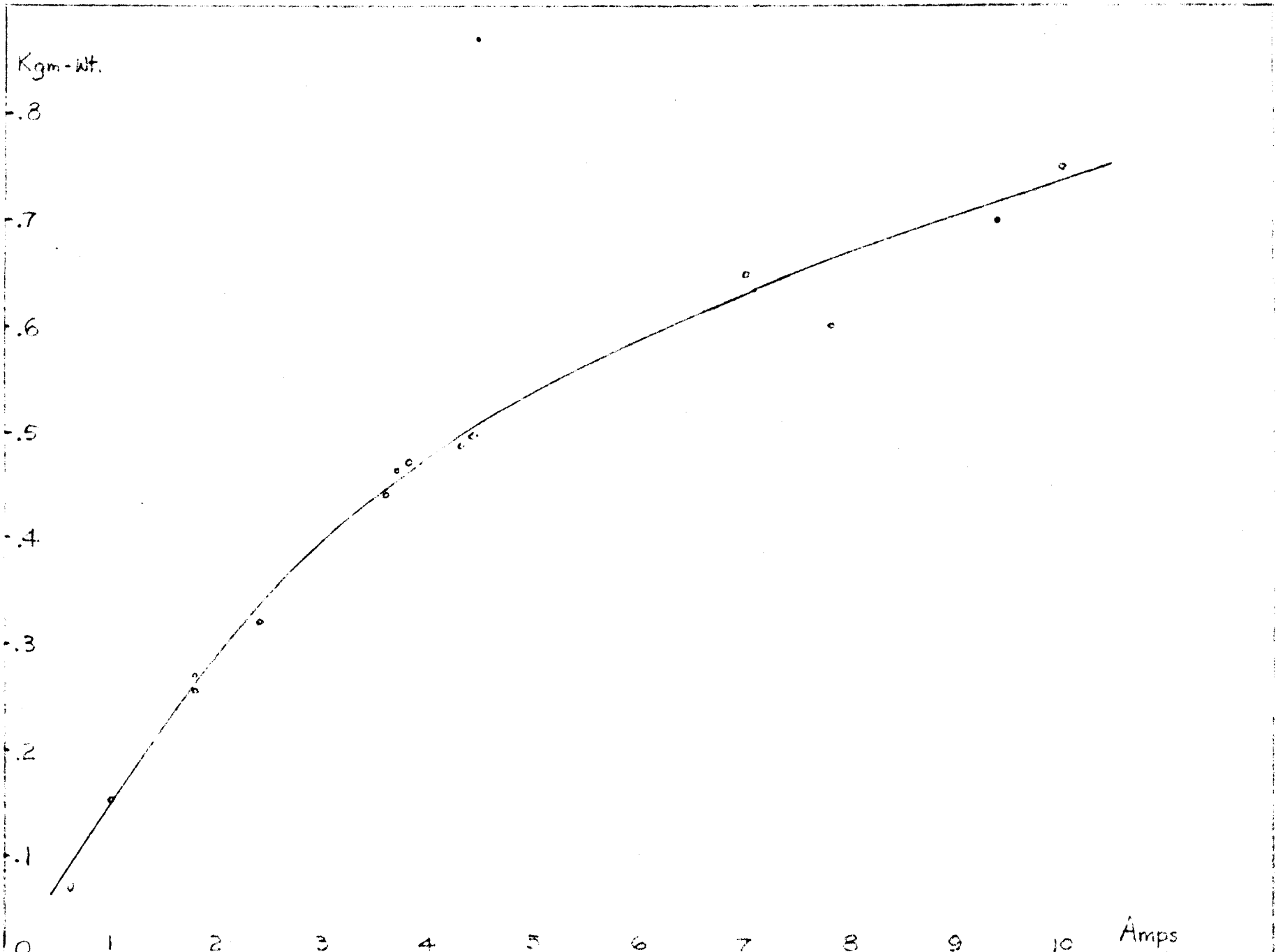

Figure 5 Load Force as a Function of Current

coil to cause separation of the samples. Separation is detected through movement of the beam from the zero reference line and through observation of the contacting surfaces by means of a cathetometer. The microbalance itself is attached to a precision linear motion feedthrough with which the balance (and upper sample) may be raised or lowered, bringing the samples into contact prior to application of the load force, and keeping them separated during bakeout.

Under optimum conditions this balance is capable of measuring forces as small as 0.1 micrograms. However, due primarily to the bucket weight, forces smaller than about 20 micrograms cannot presently be detected. This may well be suitable for the purposes of the program, but if it is not, it is possible to improve the situation by reducing the bucket weight. It is anticipated that the bucket weight could be reduced to the extent that forces as small as a few micrograms can be detected. This weight reduction would, however, reduce also the amount of load force that can be applied.

According to the manufacturer, the outgassing rate of this balance is sufficiently small so that pressures in the low 10^{-10} to upper 10^{-11} mm Hg range can be obtained with only moderate pumping. This has not as yet been verified in the present program. One pumpdown has been made with the balance in the system. A pressure of only 9×10^{-9} mm Hg was achieved. However, upon helium leak testing the system three large leaks (due to flange warpage) were found. Calculations indicate that with the elimination of these, pressures in the low 10^{-10} mm Hg range should be obtained. It is hoped that this question can be answered in the near future.

4.4 Bakeout Systems

There are two bakeout systems. The first consists of heating lamps and tapes, shielded on the outside by aluminum foil, which serve to heat the entire ultra-high vacuum part of the system up to about 150°C. The purpose of this bakeout is to aid in obtaining better vacuum. It has been found that during this operation great care must be taken to avoid any large temperature gradients as such gradients almost invariably result in the production of leaks around the copper gaskets.

The second bakeout system consists of a modified heavy-duty insulated soldering iron. This iron is placed at the same point as the electromagnet shown in Figure 4 and its purpose is to heat the samples up to high temperature (700-900°C) for surface outgassing. This system has been found to perform quite satisfactorily. However, one potential future problem is the oxidation produced upon the iron and exterior of the vacuum system. If oxidation appears to be becoming severe with usage it may be necessary to immerse the heated area (external to the vacuum system) in an inert gas, most probably helium.

5.0 SAMPLE CHOICE AND PREPARATION

Five criteria are used in the choice of sample. These are first, that the samples be representative of the more commonly occurring igneous rock and meteorite silicate minerals; second, that in so far as possible the mineral suite should encompass the igneous rock range of acidic to ultrabasic; third, that each sample be as perfect (as regards competency, purity) an example of the chosen mineral as can be obtained; fourth, that in so far as possible at least one example of each important crystal class be studied; and finally, that the sample physical properties be such that it can withstand the forming operations required in sample preparation.

A set of minerals which appear to satisfy these criteria to a reasonably good degree have been chosen. These are: orthoclase, microcline, albite, and bytownite (alternatively labradorite or anorthite) representing the feldspars; hornblende, augite, and hypersthene representing the amphibole and pyroxene groups; and epidote. Of these, samples of orthoclase, albite and hypersthene have been fabricated to date.

The sample preparation techniques are as follows. First, the crystal axes are determined and marked. This is done with the petrographic microscope when possible, and by x-ray diffraction when necessary. Cylinders of each sample, 0.5 cm in diameter and 0.32 cm long, are then cut by means of ultrasonic techniques. Intersecting perpendicular holes are then drilled, again ultrasonically (see Figure 6). A screw and lock pin are inserted into these holes to attach the samples to the experimental apparatus. The faces which are to be contacted are then polished, the polishing procedure being dependent upon the nature of the face. For perfect cleavage faces, when necessary, 400 then 800 mesh diamond grit is used followed by 5 μ garnet: for other faces this procedure is preceded by 100 then 220 mesh silicon carbide polishing. Immediately prior to use each sample is carefully washed and degreased, and oven dried. A photomicrograph is then taken of each surface.

6.0 EXPERIMENTAL PROCEDURE

The samples are first mounted, one rigidly to the lower tee flange and the other to one end of a Chatillon spring. The load force as a function of electromagnet current is determined. The upper sample is then hung at one end of a straight, five mil, tungsten wire suspended from an arm of the microbalance. A suitable counterweight (made of aluminum) is suspended from the other arm. The balance is zeroed

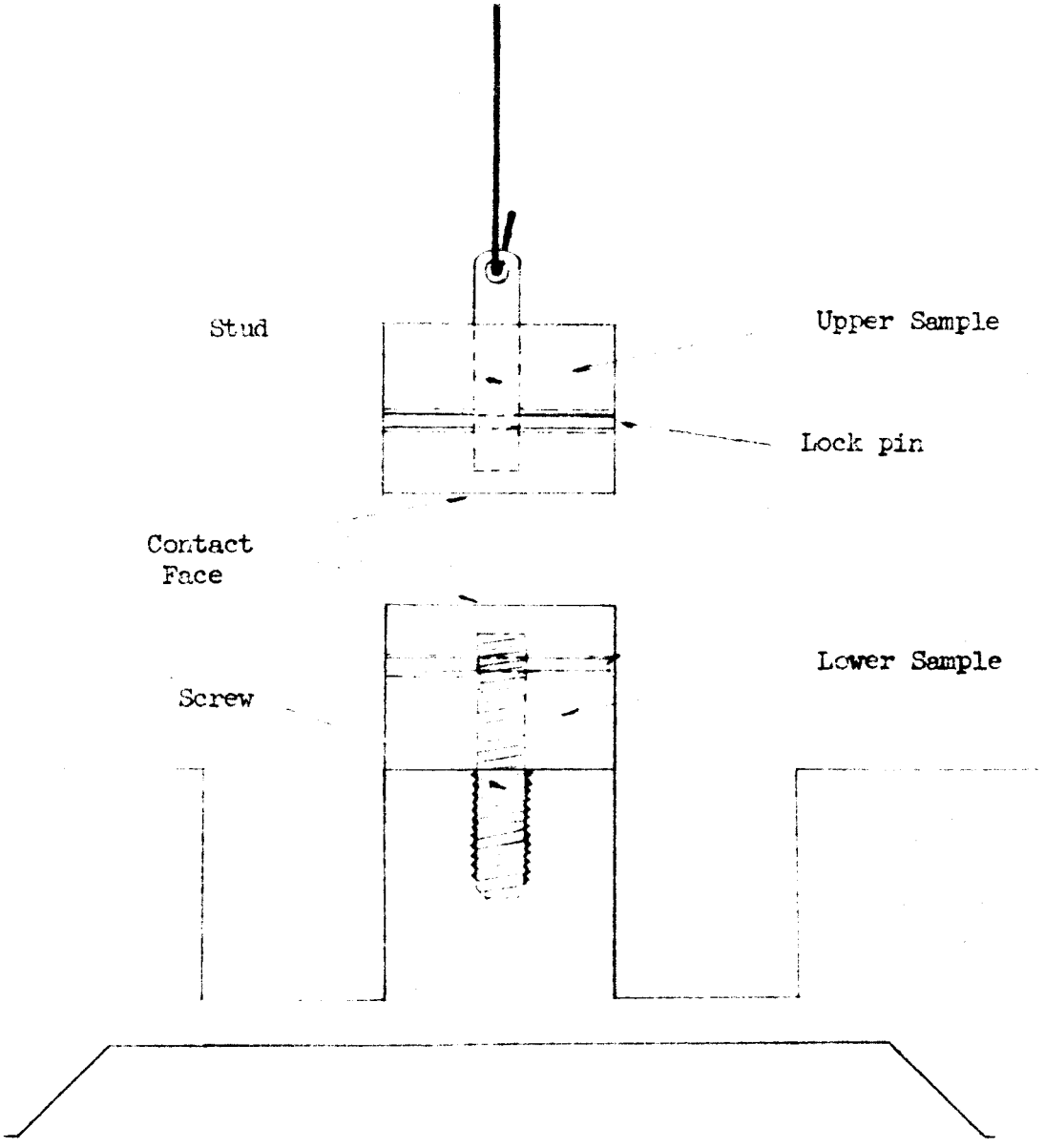

Figure 6 Sample Holder

and calibrated as per the instruction manual for the balance. The unit is then inserted into the vacuum system, the two samples aligned (and oriented with respect to crystalline structure), and the balance zero rechecked. The vacuum system is then closed, the forepump turned on and the cold trap filled. Preliminary bakeout at about 100°-150°C is performed, the temperature being monitored by six iron-constantan thermocouples placed at strategic locations, also by a thermistor attached to the microbalance. The ion pump is then turned on, the valve separating the low and high vacuum parts of the system closed and the mechanical forepump turned off. Any further bakeout necessary is performed with the ion pump on.

The samples are heated to ≈700 - 800°C immediately prior to measurement. The heating unit is then removed, the samples cooled as quickly as possible (note there are no copper gaskets in the immediate vicinity of the samples), and the electromagnet inserted (Figure 4). The upper sample is lowered into contact with the lower sample by means of the precision linear motion feedthru, care being taken that the microbalance zero is not changed. The desired load force is then applied after which the electromagnet is carefully withdrawn (to avoid residual magnetism problems). The current to the microbalance coil is slowly increased until evidence of sample separation is noted. The adhesion force can then be read directly from the microbalance control unit (located outside the chamber).

7.0 PRESENT STATUS

One attempt has been made to detect adhesion. Two orthoclase samples with contacting faces parallel to the 001 plane (the perfect cleavage plane for orthoclase) and with the faces within 10° of atomic match (in angular orientation) were used.

No load force was applied. Some indication of measurable adhesion was obtained, but the indication was marginal and any definitive statements concerning this are unwarranted at the present time. The main difficulty with this attempt was the inability to obtain sufficient vacuum, the measurement being made at a pressure of only 2×10^{-8} mm Hg.

The system was helium leak tested and three "large" leaks were found around the vacuum seals: one in the upper tee flange, one in the linear motion feedthrough, and one in the electrical feedthrough (see Figure ?). Calculations indicated that these leaks were sufficient to prevent the desired pressure from being reached. Inspection revealed that the flanges were warped, presumably during the welding (this has been a recurring problem). Replacing of the copper gaskets and the use of annealed copper gaskets did not seal the leaks. Indium gaskets were cast and these were found to work quite well. However, it was decided that copper gaskets were preferable due to the possibility of some of the indium entering the high temperature part of the system. Accordingly the upper tee flange, with associated feedthroughs, is being refabricated.

There are several unresolved questions that must be answered in the near future. First, it must be determined whether with the microbalance in the system pressures in the low 10^{-10} mm Hg range can indeed be obtained. Calculations, as noted previously, indicate this can be done and the manufacturer states it can be done. However, until this is experimentally verified in the present system it must be considered an uncertainty. Should the microbalance provide difficulties it will be necessary to semi-isolate it from the main system by means of a high impedance feedthrough tube, and to apply secondary pumping.

The second question also concerns the microbalance. During the attempt to measure adhesion noted previously, the balance, at some unfortunately unknown time during the outgassing, drifted from its zero setting. It is not presently known whether this was normal long-term drift, whether it was due to the effects of heating upon the balance mechanism (this must be considered unlikely on the basis of subsequent examination and since the balance temperature never exceeded that allowable),or whether it was due to loss of sample weight (the unbalance was in this direction). In order to circumvent this effect, assuming it may well occur again, we have replaced the originally used copper counterweights with aluminum (much lower melting point) and have shielded the aluminum from the samples. In this way it is hoped that any unbalance can be corrected through reduction of the counterweight weight by means of moderate heating. As of the present, the degree to which this may be a problem remains uncertain.

The final question concerns the magnitude of the adhesive forces. As was noted in a previous section the available experimental evidence indicates that forces significantly greater than 30 micrograms can be expected. If this is true then the adhesion measuring apparatus as it stands now has sufficient sensitivity. However, if this is not true, or more particularly not true in all cases, increased sensitivity will be required. The most feasible method for achieving this is to reduce the bucket weight. Whether or not modifications will be necessary cannot be determined until all conditions pursuant to a valid attempt to measure the adhesion are met.

8.0 SUMMARY

Work during this quarter has consisted of assembling the vacuum system, constructing the experimental apparatus, and preparing the initial silicate samples. The main problem encountered was the inability to obtain the desired vacuum due to flange warpage. It is expected that this problem will be resolved shortly.

Not all of the experimental difficulties have been resolved as yet. However, it is expected, unless unforeseen difficulties arise, to be able to proceed with the adhesion measurements shortly.

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REFERENCES

Archard, J. F., Single Contacts and Multiple Encounters,
J. Ap. Phys. 32, 1420-1425, 1961.

Buwell, E. J., Friction and Wear of Single - Crystal Sapphire Sliding on Steel,
J. Ap. Phys. 33, 2691-2698, 1962.

Dyson, J. & W. Hirst, The True Contact Area Between Solids,
Proc. Phys. Soc. Lon. B 67, 309-312, 1954.

Halajian, J. D., The Case for a Cohesive Lunar Surface Model,
Grumman Aircraft Engineering Corporation, Rept. No. ADR 04-04-64.2
June 1964.

Harper, W. R., Adhesion and Charging of Quartz Surfaces,
Proc. Roy. Soc. Lon. 231, 388-403, 1955.

Rabinowicz, E., Influence of Surface Energy on Friction and Wear Phenomena,
J. Ap. Phys. 32, 1440-1444, 1961.

Riesz, C. H. and M. S. Weber, Mechanism of Wear of Nonmetallic Materials,
Tech. Docum. Rept. No. WADC-TR-59-316, Armour Research Foundation,
June 1962.

Roshon, D. D. Jr., Selected Area X-Ray Diffraction Studies of Surface Damage
in Sliding on Single - Crystal Copper,
J. Ap. Phys, 35, 1262-1269, 1964.

Spalvins T and D. V. Keller, Bulk Adhesion, in Adhesion Between Atomically
Clean Metallic Surfaces, by D. V. Keller, Syracuse University Research
Institute Rept. No. MET. E. 905-621 SA, Jan. 1962.

Salisbury, J. W., P. E. Glaser, B. A. Stein and B. Vonnegut,
Adhesive Behavior of Silicate Powders in Ultrahigh Vacuum,
J. Geophys. Res. 69, 235-242, 1964.

REFERENCES (continued)

Steijn, R. P., Sliding and Wear in Ionic Crystals,
J. Ap. Phys., 34, 419-428, 1963.

Stein, B. A. and P. C. Johnson, Investigation of Soil Adhesion
Under High Vacuum, in The Lunar Surface Layer,
pp 93-105, Academic Press, New York, J. W. Salisbury and P. E. Glaser
eds., 1964.

Stone, W., Some Phenomena of the Contact of Solids,
Phil. Mag., 9, 610-620, 1930.

Tomlinson, G. A., Molecular Cohesion,
Phil. Mag., 6, 695-712, 1928.

Tomlinson, G. A., Further Experiments on the Cohesion of Quartz Fibres,
Phil. Mag., 10, 541-544, 1930.

Walton, O., Mechanism of Friction,
J. Ap. Phys. 33, 519-526, 1962.